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Cables :: "Stomata." | Phone :: 0365

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THE RISE OF ROTENONE *

ROTENONE is one of the most potent insecticides in use today. For 300 years, it was known only to aborigines who used it to poison and capture fish. As recently as 1930, trade in the roots of rotenone-bearing plants was practically non-existent, yet in 1940 the United States alone imported 6½ million pounds in crude and powdered form. It is one of the most astonishing developments in agricultural history.

Centuries before we learned that plant poisons could be used to kill insects that prey on human food, the natives of the American, Asiatic, and African tropics were using the same plants to stun fish and thus solve more directly the problem of keeping their stomachs full. Today, of course, the use of poison to catch fish has been made illegal in most countries on the theory that it is harmful to wildlife, but use of the plants in killing insects has made them more valuable than ever before. The actual poison consists of rotenone and substances related to it called rotenoids. It is a colourless, crystalline compound, which in powdered form appears white. It is one agricultural product that seems definitely safe from synthetic competition, since the chemical formula, while known, is so complex that there is little possibility that it will ever be duplicated in a test tube.

Rotenone is found as a solid in the dried roots of certain plants of the bean family which are native to most tropical sections of the globe. Some produce negligible amounts of rotenone and some produce as much as 12 per cent. What might at first seem a complex relationship is simplified by the fact that there are only two important rotenone-bearing branches of the family—the Derris of the Far East and the Lonchocarpus of Latin America. The cube (pronounced COO-bay) of Peru and the timbo' of Brazil produce most of the Latin American rotenone of commerce today. The haiari and the nekou of the Guianas are minor sources. The Spanish-speaking people of Latin America generally call all fish-poisoning plants barbasco, whether they contain rotenone or not.

The discovery that some of the fish poisons are also potent insecticides was probably made centuries ago, but there was no reliable information

* Abridged from an article by R. C. Roark in *Agriculture in the Americas*, Vol. I, No. 9.

on the subject until comparatively recently. In 1848, the use of derris as an insecticide in British Malaya was officially reported, but the Chinese had used it there a long time before to kill insects attacking growing vegetables. In 1909, the botanist Bryant found African Zulus killing head lice with an extract made from the leaves of certain species of Tephrosia, a relative of Derris and Lonchocarpus. In 1910, cube was used as an insecticide to destroy ticks on llamas in Peru.

Following the early accounts in England of the use of derris as an insecticide in British Malaya certain chemical manufacturers there began the preparation of liquid insecticides containing derris extracts. These products, which were sold under trade-mark names, appeared on the market in England in 1911 and shortly thereafter in the United States. For the most part they were recommended for use in controlling aphids and insects affecting animals, especially lice. Even for these pests the first insecticides made from the roots of derris failed to have extended use, largely because they were variable and their effectiveness was uncertain.

Not until 20 years later, after extensive chemical research, were manufacturers able to go ahead and make derris preparations with the assurance of producing standardized insecticides that would be effective. The urgent need for an insecticide which would not leave objectionable residues on the marketed product encouraged entomologists to make extensive tests of derris, and its value for the control of certain plant-feeding insects was soon established. Its popularity grew rapidly and from 1932 to 1940 the importation of derris root into the United States increased from 38,000 pounds to 3,220,972 pounds, or 84 times!

Today, the potency of rotenone against many pests is firmly established. It is 15 times as toxic as nicotine when sprayed upon bean aphids and 25 times as toxic as potassium cyanide to gold-fish. There is, however, a wide variaton in its effect on different insects. Mosquito larvae succumb to 5 parts of rotenone in 1,000,000 parts of water. The southern armyworm, on the other hand, eats the substance with relish, or at least with no ill effects, and several species of borers attack the dried derris and cube roots in storage, reducing the material to powder and greatly reducing its value.

It is an ideal insecticide in many respects because it is highly toxic to most cold-blooded animals and relatively non-poisonous to warm-blooded animals—30 times as poisonous as lead arsenate when fed to silkworms but only one-thirtieth as toxic as the same substance when fed to rabbits, for instance. That is good news, not only to rabbits, but also to human beings, for it means that cabbage, cauliflower, peas, beans, and other foodstuffs can be treated with rotenone without the slightest harmful effect on consumers.

While entomologists have tested rotenone insecticides upon almost 800 different kinds of insects and have found them effective against many species, their use is as yet recommended only against comparatively few. Further study probably will extend their use. Even now, however, rotenone dusts and sprays are in large-scale use for the control of many pests of outstanding importance, including the pea aphid, the pea weevil, the Mexican bean beetle, cabbage worms, fleas, flies, lice and ox warbles.

In the natural state, rotenone-bearing plants are large woody climbers which up to the age of 4 years have a bushy form and later may reach the tops of tall trees. When cultivated in plantations the roots are customarily harvested when the plants are 2 or 3 years old. Tropical conditions with not less than 80 inches of rain a year are necessary for their growth. In the principal producing countries Lonchocarpus is cultivated on plantations of various sizes, often in the vicinity of dwellings. In Peru, planting is usually done in September or October, and the plants reach a height of 3 feet the first year, 5 to 6 feet the second year, and perhaps 8 feet the third year.

One difficulty in cultivation is the heavy growth of weeds. This is reduced by interplanting with such crops as cassava, beans, and pineapples, a practice that also permits more economical use of the land. The root is harvested at the end of the second, third, or fourth year, usually in the dry season when the work is easier. Data on yields and rotenone content are unreliable, but they are sufficient to indicate that under proper conditions the crop is profitable for growers.

Cube, timbo', and derris are imported into the United States chiefly in the form of dried roots, from which the powdered commercial insecticides are produced. The crude roots are finely ground in special mills and then mixed with clay or talc to a rotenone content of 1 per cent. or less. Most of the dusts on the market contain only about three-fourths of 1 per cent. rotenone. If thoroughly applied, dusts containing as little as one-fourth of 1 per cent. are effective against the pea aphid, cabbage worms, and many other insects. Extracts from these roots dissolved in safrol, alkyl phenols, or other solvents are added to kerosene to make fly sprays. Pyrethrum extract is often added to cube extract, as the combination is especially effective.

The use of all rotenone-bearing plants as insecticides is bound to expand. Rotenone and the rotenoids associated with it are effective against a wide range of agricultural as well as household insects, except in extreme concentration they are harmless to man and domestic animals, they are cheap, they can be applied safely to the most tender vegetation and they leave no poisonous residue on fruits and vegetables.

The Editor is prepared to consider signed articles for publication in *Tropical Agriculture*. Such articles should normally vary between 1,000 to 6,000 words and may be illustrated if desired.

THE ZONES OF SUPERSATURATION IN RAW CANE SUGAR LIQUORS

Their Determination and Influence on the Crystallization of Sucrose

J. G. DAVIES AND R. D. E. YEARWOOD
(*Department of Sugar Technology, Imperial College of Tropical Agriculture, Trinidad, B.W.I.*)

(SYNOPSIS.—*A previously published method for rapidly estimating the solubility data of sucrose in impure solutions has been used to define the limits of the different zones of supersaturation in raw cane sugar factory liquors. In the 60 to 80 purity range, the upper limit of the metastable zone appears to lie between supersaturations 1.25 and 1.55; it varying inversely with the purity. These values are higher than those recorded in the literature. The intermediate zone, as defined by Webre, appears to be so narrow as to be of little practical importance. Grain will form by the spontaneous or the shock method in the labile zone. By either method, increasing the initial supersaturation results in an increase in the crystal content after one hour.*)

Introduction

Claassen first made the observation that in the control of the crystallization of sucrose, the degree of supersaturation of the mother liquor is of more practical importance than the degree of concentration. As far as the cane sugar industry is concerned few attempts have been made to determine the sucrose solubility data for factory liquors. Without this information it is therefore impossible to know what solubility relationships exist at any one stage of the process, and it is consequently impossible to determine the optimum conditions for operation.

The investigations reported herein were started by developing a rapid and accurate method for determining the solubility of sucrose in impure solutions (1). The method was then applied to syrups and molasses of varying purities from the Experimental Sugar Factory attached to this Department. During the course of the later experiments, the limits of the zones of supersaturation have been defined and the influence determined of varying supersaturation values upon the formation of nuclei and the deposition of sucrose.

Theories of Granulation

On the basis of Ostwald's phase rule theory of crystallization, Webre (5) defines three zones of supersaturation in sucrose solutions as follows: (i) *The metastable zone*, extending from the saturation line to the metastable line. Within the zone so formed, the conditions are such that sucrose deposition will take place on existing crystals, but no new nuclei will form. (ii) *The labile zone*, which includes the higher values of supersaturation; in this zone the conditions are such that new crystals will form spontaneously. (iii) *The intermediate zone*, which lies between the metastable and the labile zones; here the conditions are such that fresh nuclei will form only in the presence of other solid particles of sucrose.

According to these definitions, the metastable zone is the one used in practice when sucrose is to be deposited on existing crystals. This occurs during the "building" stage of the boiling process, that is while established crystals are being grown to a more desirable size. The metastable zone is also the one employed in the Webre seeding system (6). The intermediate zone is, according to Webre, utilized for grain formation by the shocking method, in which a very small quantity (100 to 200 grams) of sucrose fragments is introduced to the boiling syrup or molasses. The shock thus effected results in the rapid formation of fresh nuclei. Operators tend toward the opinion that a more even granulation is produced by this method than by spontaneous granulation. The labile zone must be established before a liquor will form grain spontaneously. Production of grain by this procedure is known in the industry as the waiting or spontaneous method.

While stipulating that the values of supersaturation defining the boundaries of the zones will vary according to the purity of the liquor, Webre (5) quotes the following for pure sucrose solutions:

TABLE I

Zones of Supersaturation in Pure Sucrose Solutions (Webre)

Supersaturation Zone	*Supersaturation Value*	*Remarks*
Labile	Above 1.30	Used for graining by waiting method
Intermediate	...1.20-1.30	...Used for graining by shocking method
Metastable	...1.00-1.20	...Used for seeding method

Methods, Equipment and Procedure

In the experimental work described herein, the following methods of analysis were employed:

(i) °Brix, by the Zeiss Refractometer (28°C) at first, but later the more convenient Bellingham & Stanley Projection Refractometer (20°C) was adopted.

(ii) % Sucrose, by Jackson & Gillis Modification No. 4.

(iii) Purity, Sucrose % Refractometer Brix (Suc./Refr.)

(iv) Crystal Size, by measurement of the image projected by a Shadowgraph, later by measurement of photomicrographs.

(v) Crystal Weight & Surface area, by the use of Thieme's tables (3).

(vi) % Conglomerate, by counting.

(vii) Solubility data, by the evolved method (1).

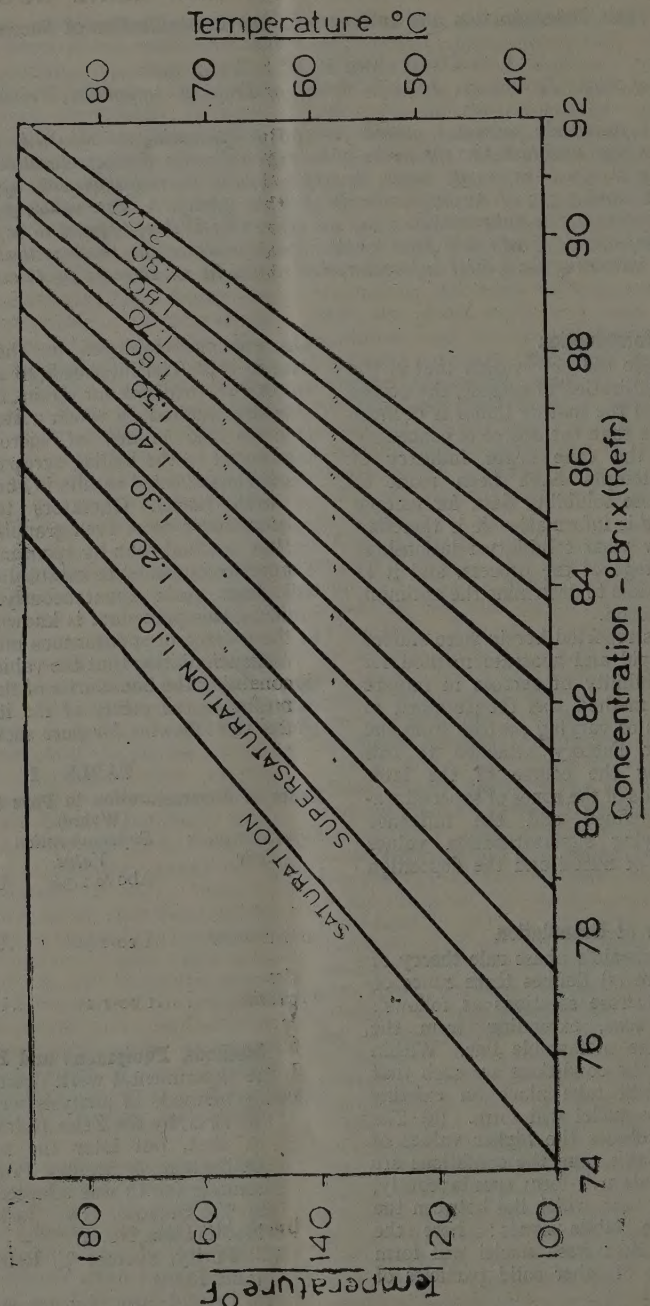

FIG. 1.—Sucrose solubility chart for % sucrose/ refractometer purity I.C.T.A. molasses. Crop 1943.

The experiments were conducted both in the laboratory and in the Experimental Sugar Factory. The procedure was to store a bulk sample of a suitable syrup or molasses and then to determine its sucrose solubility data (1). Once the temperature-saturation-concentration relationships had been established, it was a matter of simple calculation to determine the concentration for any desired supersaturation at any of the working temperatures. For working purposes, the concentrations were calculated from supersaturation 1.00 to 2.00 at intervals of 0.1 at four different temperatures and then charted. A typical chart is reproduced in Figure 1. It will be noticed that the saturation and the different supersaturation lines are straight. To produce a liquor at a desired supersaturation, it is merely necessary to observe the working temperature and then to read off from the chart the required concentration as °Brix (refr.).

In the laboratory, concentration was conducted in *vacuo* in a 12-litre round bottom Pyrex flask heated by a water bath. At the desired point, the interior of the flask was brought to atmospheric pressure, the flask was removed from the bath and the molasses transferred to a closed water-jacketed container maintained at the same temperature as the boiling molasses. The temperature of the container and the molasses was regulated by the circulation of water from a thermostat tank. The stirrers in the container could be operated at 1, 5 or 25 r.p.m.

In the Experimental Factory, the material was concentrated in *vacuo* in a 56 cu. ft., 4 ft. 6 in. diameter coil vacuum pan. At the desired concentration, it was struck to a clean, open 40 cu. ft. lagged magma-mixer the stirrer arms of which revolved at 22 r.p.m. The stirrers are of the common paddle type used in ordinary factory crystallizers.

All the experiments were started with a molasses of known purity. As nuclei formation occurred and sucrose deposition proceeded, the purity of the mother liquor decreased. But since only pure sucrose had been deposited, a refractometer reading of the concentration of the mother liquor at any stage of the experiment enabled the purity of the mother liquor to be calculated. The degree of supersaturation could then be read off from the appropriate chart. The method for calculating the purity of the mother liquor was as follows: the purity of the massecuite remains constant during treatment. Therefore a determination of sucrose % massecuite enables the concentration of the massecuite to be calculated. An estimate of the impurities % massecuite is obtained by difference (Impurities by refractometer=°Brix (refr.)—% sucrose). Theoretically, the impurity/water ratio of a massecuite is equal to that of its mother liquor. Therefore, from an observation of the °Brix (refr.) of the mother liquor, the water content of the mother liquor is obtainable (Water by refractometer=100—°Brix (refr.)), thus the impurity content (Impurities=Water × Impurity/Water ratio) and likewise the sucrose % mother liquor (% Sucrose=°Brix (refr.)—%Impurities).

Several methods were tried in an attempt to separate the small grain from the mother liquor, so that the purities could be obtained by analysis. But the attempts failed and thus resort had to be made to the use of the impurity/water ratio method. Although there is reason to suppose that this method yields exaggerated results, they are nevertheless believed to be comparable. The method is simple and allows of many more observations being made than if a separation method had been employed.

The Metastable Zone of Supersaturation

By definition, this is the zone in which no new nuclei will form. Hence the method of approach was to observe the highest supersaturation at which no grain formation would take place. The observed value would then lie on the upper limit of the metastable zone. The observations were made in the Experimental Factory using materials of three different purities. It will later be shown that the intermediate zone as defined by Webre appears to be of little practical importance and once the supersaturation exceeds the upper limit of the metastable zone, nuclei will form either by shock or spontaneously. When the liquor is maintained at constant water content and constant temperature, the rate of formation of nuclei is accelerated by stirring. The procedure therefore was to concentrate the molasses in the pan and discharge it to the lagged magma-mixer. Increasing degrees of supersaturation were used in successive runs at the same purity. Samples of the material were then examined on a slide under the microscope (300 ×) at frequent intervals for the presence or absence of sucrose crystal nuclei. For example, at 60 sucr./refr. purity and supersaturation 1.57, a few crystal nuclei appeared after some minutes. But on maintaining the supersaturation at 1.50, no nuclei could be detected after 25 mins. One pound of shock grain was then added. No effect was observed. Hence the upper limit of the metastable zone for 60 purity molasses lies between supersaturations 1.50 and 1.57 and it was recorded as supersaturation 1.50 at 158°F.

The results for this and the other purity materials are shown in Table II.

TABLE II

Upper Limit of the Metastable Zone at Different Purities

Purity	*Supersaturation at upper limit*
60.0	1.55
70.0	1.30
80.0	1.25

The supersaturation at the upper limit of the metastable zone decreases with increase in purity. The results are presented in graphical form in

Figure 2. It is of interest to note that as far as 60 sucr./refr. purity is concerned, the observed values far exceed those quoted by Webre (5), Mayoral (2), Thieme (4) and others.

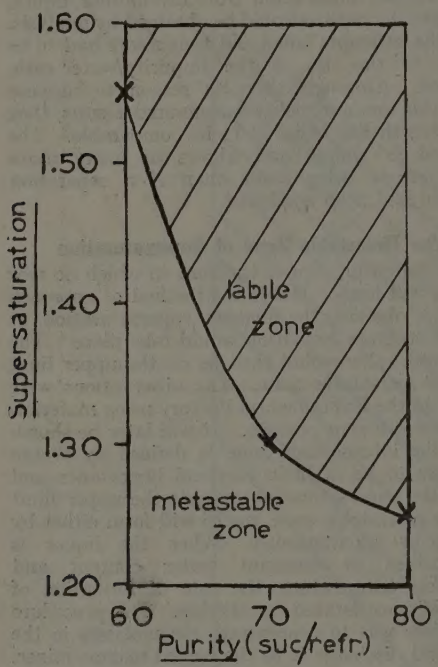

FIG. 2.—Supersaturation purity relationships at the upper limit of the metastable zone.

In the 60 sucr./refr. purity series, the opportunity was taken to test the effect of temperature on the degree of supersaturation which defines the upper limit of the metastable zone. The results are tabulated in Table III:

TABLE III

Effect of Temperature on the Upper Limit of the Metastable Zone 60 Purity

Temperature °F.	*Supersaturation at upper limit*
141	1.55
158	1.50
172	below 1.50

The effect of temperature does not appear to be very great, and within the normal range of operating pan temperatures it is negligible. It may however be of importance during crystallization-in-motion, when the change in temperature is of the order of 50°F.

In practice, whether seed is being established or established grain being grown, it is obvious that the operation should be carried out as near to the upper limit of the metastable zone as conditions will allow. In the sugar factory, the maintenance of the boiling conditions in such a delicate state of equilibrium would require the installation of adequate and properly designed control instruments and a mechanically operated stirrer to ensure free circulation of the massecuite. This equipment is expensive, but it might be justified if it could be shown that the rates of sucrose deposition are appreciably increased when the mother liquor is accurately maintained at a supersaturation near to the upper limit of the metastable zone.

A series of experiments was therefore conducted in the laboratory in which the seed rate, temperature, rate of stirring and purity of mother liquor were constant, and the degree of supersaturation of the mother liquor was varied. The sucrose deposition was then calculated at the end of one hour. The results are presented in Table IV.

TABLE IV

Effect of Supersaturation on the Rate of Sucrose Deposition

Run No.	1	2	3
MOTHER LIQUOR			
Supersaturation ...	1.45	1.50	1.55
Temperature °F ...	140	141	141
Suc./refr. purity ...	60.60	60.80	61.20
SEED RATE			
Icing sugar % by wt	5.5	5.5	5.5
Estimated no. of crystals, 1000's ...	10500	10500	10500
RATE OF STIRRING			
r.p.m.	25	25	25
AFTER 1 HR.			
Temperature °F. ...	140	142	142
Supersaturation ...	1.26	1.28	1.28
Sucrose deposition ... (gr. deposited/ 100 gr. massecuite /hr.)	7.49	7.91	9.25

The deposition rates are shown graphically in Figure 3. It would appear to be well worth while in practice to maintain the mother liquor as near to the upper limit of the metastable zone as possible. At supersaturation 1.55, 23.5 per cent. more sucrose is deposited at the end of 1 hour than at supersaturation 1.45.

The Intermediate Zone of Supersaturation

By Webre's definition (5), the intermediate zone is bounded on its lower limit by the metastable zone and on its upper limit by the labile zone. Also, it is the zone in which the formation of sucrose crystal nuclei can be induced by shock but in which they will not form spontaneously.

It will have been noticed that in the experiment described above which defined the upper limit of the metastable zone for 60 purity material as supersaturation 1.50, a few crystal nuclei formed spontaneously at supersaturation 1.57. Hence, if the intermediate zone does in fact exist, it must lie between the supersaturation values of 1.50 and 1.57.

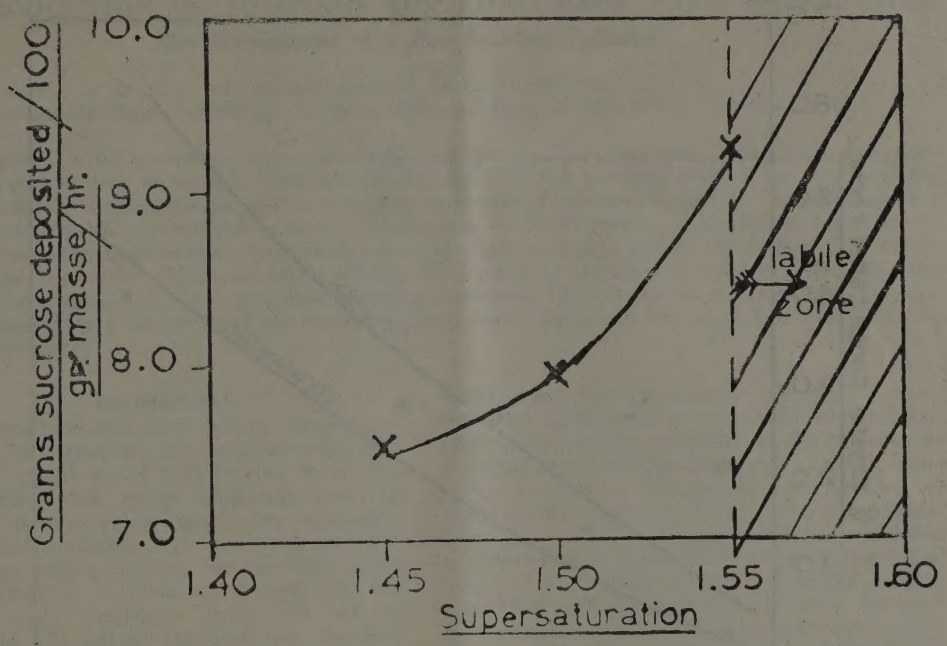

FIG. 3.—Effect of supersaturation on the rate of sucrose deposition in the metastable zone. 60 sucrose/refractometer purity.

Reference to Figure 2 shows that at 60 purity the metastable zone covers a much wider band of supersaturation values than it does at the higher purities. It is logical to suppose that a similar condition would exist in the intermediate zone. Yet at 60 purity, the band of supersaturation values defining the intermediate zone is at the most only 0.07 degrees in extent.

It is therefore concluded that the intermediate zone as defined by Webre is so narrow in extent as to be of little practical consequence. Therefore the zones of supersaturation of importance in commercial sugar boiling are the metastable zone, in which sucrose can only be deposited on existing crystals, and the labile zone, in which fresh nuclei may be formed either spontaneously or by shocking.

The Labile Zone of Supersaturation

The first series of laboratory experiments conducted in the labile zone were designed to show the effect of the supersaturation of the mother liquor at graining upon the crystal content after one hour's stirring at constant water content and constant temperature. The procedure was to concentrate 60 purity molasses to the pre-determined supersaturation value. Half was then transferred to one container and the other half to a second container, both of which were maintained at the temperature of the molasses. Both sets of stirrers were operated at 25 r.p.m. To the molasses in one container, 0.05 grams of icing sugar were added as shock, while the molasses in the other was allowed to grain spontaneously. In order to ensure the use of grain-free molasses only, a sample was taken during the transfer and examined on a slide under the microscope (300 ×). The crystal content was calculated by Deerr's well-known s-j-m formula. The temperature used was 141 °F. The analytical results are shown in Tables V (*a*) and V (*b*) and the crystal contents are graphed in Figure 4.

TABLE V(a)

Effect of Supersaturation on Crystal Content after One Hour. Spontaneous Granulation. 60 Purity

Run No.	1(*a*)	2(*a*)	3(*a*)	4(*a*)	5(*a*)
Initial Supersaturation	1.65	1.80	1 90	2.00	2.30
Initial Purity ...	60.60	60.60	59.57	59.60	59.60
Final Purity (1 hr.)	60.30	59.95	58.48	56.86	55.67
Per cent. Crystal Content (1 hr.)	0.33	0.68	1.13	2.82	4.02

TABLE V (b)

Effect of Supersaturation on Crystal Content after One Hour. Shock Granulation. 60 Purity

Run No.	1(*b*)	2 (*b*)	3(*b*)	4(*b*)	5(*b*)
Initial Supersaturation	1.65	1.80	1.90	2.00	2.30
Initial Purity ...	60.60	60.60	59.37	59.60	59.60
Final Purity (1 hr.)	58.62	59.24	56.97	56.07	53.45
Per cent. Crystal Content (1 hr.)	2.08	1.42	2.68	62.3	6.26

Under the conditions of these laboratory experiments it appears that the addition of a small amount of shock is beneficial. It results in greater depositions of sucrose, the footings are more attractive from a factory point of view, though they are not free from conglomerate. Examination of the samples further revealed that

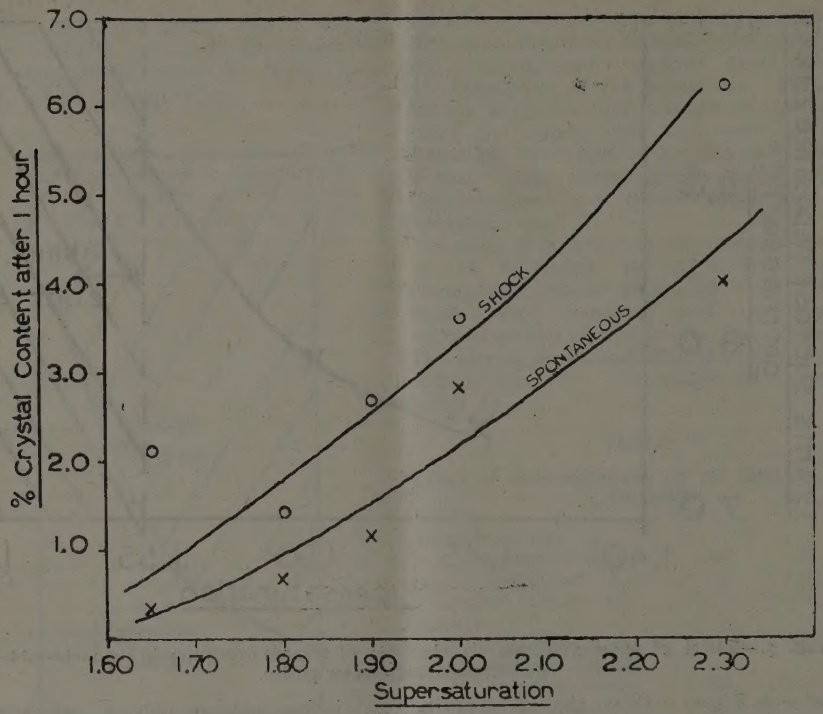

FIG. 4.—Effect of supersaturation on the crystal content after 1 hour's stirring. Labile zone. 60 sucrose/refractometer purity.

there is no optimum supersaturation for graining spontaneously or by shocking, the results becoming increasingly better with increase in the initial supersaturation. Hence it follows that in graining by either method in the factory, the best supersaturation to use is the highest obtainable.

In the Experimental Factory, molasses of 60 purity was grained in the described equipment by both methods. The results confirm the laboratory ones, with the exception that under the conditions of better circulation obtaining in the factory the spontaneous method appears to be as good as or better than the shocking method. An entirely new graining technique has now been worked out which seems to possess great practical possibilities. It is described in another paper, in which some further data are presented.

A fact of some interest that was observed in all the experiments, whether the material was grained spontaneously or by shocking, was that nuclei formation was first made manifest by the appearance of one or two crystals only. They could be readily seen under a magnification of 300 ×. There was then an appreciable time lag during which the number of crystals slowly increased. After that, the sudden formation of a tremendous crop of new nuclei occurred which was simultaneously accompanied by a rapid acceleration of the rate of crystal growth.

If the secondary granulation is due to the presence of the first few crystals which appeared, then the addition of shock should bring about a vast crop of crystals immediately. But under the conditions of constant water content and constant temperature, this has not been observed to occur.

The phenomenon has been noted at each of the three purities examined. At constant supersaturation, the time lag decreases with increase in purity. At constant purity, the time lag decreases with increase in supersaturation. Similar behaviour has been recorded when molasses from other factories in Trinidad and Barbados have been grained in the pan. No tenable explanation has yet been found.

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THE PRODUCTION OF FOOTINGS FOR LOW GRADE CANE MASSECUITES

The Development of a New Graining Technique

J. G. DAVIES AND R. D. E. YEARWOOD
(*Department of Sugar Technology, Imperial College of Tropical Agriculture, Trinidad, B.W.I.*)

(SYNOPSIS.—*Webre's seeding system of establishing footings for low grade massecuites has been critically examined both in the laboratory and the factory. A new graining method has been developed which is conducted at constant water content and under conditions of vigorous circulation. A simple stirring vessel separate from the vacuum pan is used. Molasses of 60 to 70 purity can be grained satisfactorily, depending upon the requirements. Crystallization occurs spontaneously with the production of a highly desirable type of crystal. After one hour's stirring the crystals are regular, of average size 0.18 mm., and free from conglomerate. The pan floor operating features of Webre's system are retained. The new technique, now taken as far as the Experimental Factory stage, appears to have important possibilities in commercial practice.*)

Introduction

All modern raw cane sugar boiling systems are based on the formation and establishment of a footing of crystal nuclei, the working up of the footing into a low grade massecuite and the mixing of the low grade sugar, after separation from the final molasses, with raw syrup or "A" molasses to form a footing for the high grade strikes. Thus, in a balanced system, all the shipping sugar originates from one set of granulations. It follows therefore that the ease of operation, degree of exhaustion, rate of curing and the other factors of vital practical importance are to a lesser or greater extent dependent upon the skill with which the original processes of crystal formation and establishment are carried out.

There are two recognized methods of forming fresh crystal nuclei within a suitable mother liquor. They are: (i) by the waiting or spontaneous method, and (ii) by the shocking method. At one time it was thought that while the spontaneous process demanded the creation in the mother liquor of the labile zone of supersaturation, a shock-granulation could be conducted within the lower, intermediate zone. But recent work (1) in these laboratories has shown that in practice both take place within the labile zone and that the intermediate zone appears either not to exist or to be so narrow in extent as to have little practical significance.

The chief practical disadvantage in operating either of the two methods of forming fresh nuclei is the variation which exists, even among skilled pan boilers, in judging the desirable number of crystals. It is difficult to devise a means suitable for use on the pan floor which would result, with some degree of assurance, in the check charge of water or liquor being taken at the correct time. Casual examination of a sample, under the low-powered magnifier with which most pan floors are equipped, does not reveal the immense variations which are tolerated and described as "normal" granulations. Probably one of the reasons for this is that while concentration is proceeding in the vacuum pan during the actual process of grain formation, the conditions affecting the solubility of sucrose, such as water content and perhaps temperature, are constantly changing. Also, there are differences in the sucrose solubility relationships during the period of the crop due to changing ash, reducing sugars and organic non-sugar contents of the liquor. Factors such as these have a profound effect on the granulation. Thus the process may appear to the pan boiler to proceed quite normally in so far as the liquor feels and looks quite normal, the steam pressure is normal and the vacuum is normal. He may be confident that he has introduced the check charge at the right time. But the fundamental factors are against his being able to reproduce consistently the same number of crystals per unit volume.

Webre (5) realized the short-comings of these two methods and evolved a new system. In it, the mother liquor, after the metastable zone of supersaturation has been established, is seeded with a quantity of fine fragments of sucrose crystals in the form of 6 × icing sugar. It follows that if the metastable zone is then maintained, no new crystal nuclei can be formed and deposition must take place on to the introduced fragments. When this occurs, the fragments grow and form true crystals. The usual procedure for establishing and growing young grain is followed from then on. The advantage of this system is that since each of the crystals in the final massecuite originated as an introduced fragment, the number of fragments controls the number of crystals. Hence the number, and therefore the size, of the final crystals can be varied at will. This supposes that at no stage during the subsequent boiling do any fresh crystals, or smear, form in the massecuite. It is a condition which very seldom obtains but it can be very materially reduced by the use of pans of proper design with or without mechanical circulation.

Until recently, the high purity raw syrups (80 to 88 purity) were used for graining by the spontaneous or the shock method. It forms the basis of the Java system of boiling. Its operating disadvantage is that syrup, from which no sucrose has yet been recovered, forms part of the low purity exhaustion strikes. The latter are usually boiled to 55—57 apparent purity. Thus the molasses which form the other part of the low

purity strikes has to be exhausted to a still lower purity so that, on mixing with syrup, the predetermined purity of massecuite is obtainable. This involves unnecessary boiling in the pans and, usually, excessive re-circulation of solids in the effort to control purities.

Webre's seeding system also overcomes this difficulty because the mother liquor to be seeded is 60 purity "A" molasses. No syrup need be by-passed direct to the exhaustion strike. He further claims that by the use of his system, the conglomerate content of the final sugar is materially reduced.

Examination of Webre's System

Webre's system of seeding 60 purity molasses has been examined critically both in the laboratory and in the Experimental Factory. The same equipment and methods were used as those described previously (1). Since the entire boiling process in this system is carried out in the metastable zone of supersaturation, the factors examined were the effect of varying seed rate and the effect of circulation. The latter is of some importance. In the original specification of the system, Webre (5) stated that for half an hour after the introduction of the seed, the steam is turned off but the mechanical circulator is operated to ensure adequate movement of the fragments in the mother liquor. The reason for this is that while the number of nuclei is very large, the available surface area is very small. Nothing that can affect the solubility relationships should therefore be disturbed until more surface area is available for the deposition of sucrose. The growth of the fragments takes place at the expense of sucrose in solution in the mother liquor. It follows that the degree of supersaturation must therefore decrease during this period since there is no evaporation or temperature change to maintain it.

Seed Rate

For ease of manipulation, the first series of laboratory experiments was conducted using through 35, on 48 (Tyler's standard screens) screened and dried granulated sugar. The average grain size was 0.375 mm. In each case, the mother liquor was concentrated to a metastable zone value of supersaturation 1.45, as indicated by the determined solubility chart. Varying seed rates were tested. The stirrers were set at 25 r.p.m. The results are presented in Table I.

TABLE I

Effect of Seed Rate. 60 Purity Molasses

Seed Rate % Molasses	*Mother Liquor Supersat.* After ½ hr.	After 1 hr.	After 20 hrs.	*Grams Sucrose deposited per Gram Seed after First Hour*
15.0	1.40	1.38	1.25	0.236
20.0	1.33	1.28	1.22	0.272
25.0	1.33	1.28	1.26	0.210

The first observation to be made from these results is that even at the highest seed rate (25%) and after prolonged stirring (20 hours) the deposition of sucrose from 60 purity molasses in the metastable zone proceeds very slowly once the supersaturation of the mother liquor has fallen to a value of about 1.27. It follows then that when the process is applied in commercial practice, the initial supersaturation must be such as to ensure that this value is not attained during the stirring period. For this size of seed, which is considerably larger than that used in practice, the optimum seed rate is 20 per cent. by weight on molasses. At this rate, the amount of sucrose deposited per unit weight of seed is at or near the maximum.

The series was next repeated using two smaller sizes of seed. The necessary amount of each was determined which would yield the same result of reducing the supersaturation of the mother liquor from 1.45 to 1.27 in one hour of stirring. The total surface area available at the determined optimum seed rate for each size of seed together with an estimate of the rate of crystallization could then be calculated. The results are presented in Table II.

The optimum rate, the surface area and the number of nuclei are each expressed on the basis of 100 grams of molasses. The rate of crystallization is the grams of sucrose deposited per square cm. of surface area per 100 grams molasses per hour.

As the size of the seed decreases, so the optimum percentage by weight of it also decreases. This is to be expected since the surface area/weight ratio of particles increases as the size decreases.

On the basis of surface area, however, it is seen that to obtain a given performance more area is required in the smaller sizes of seed than in the larger ones. Furthermore, the rate of crystallization or efficiency per unit of area also decreases. This observation may be explained by (*a*) the unsuitability of applying Thieme's tables to fragments instead of whole crystals, (*b*) the fact that small fragments tend to form conglomerates very easily, and hence the total surface area is not effective, and (*c*) consideration of the settling rates of different sized crystals in molasses. The third possibility is of some interest. A small crystal will fall through a column of liquid at a slower rate than a large one. Hence when the molasses is in motion, as in a vacuum pan or the laboratory container, a small crystal will come in contact with new zones of mother liquor less frequently than will a large one. This would result in sucrose being deposited on to a large crystal at a faster rate, which is in accordance with the observation.

A value known as the Effective Surface Area (E.S.A.) was then calculated. It is based on the assumption that the area of through 35, on 48 seed is 100 per cent. efficient because the efficiency increases with the size and it is far larger than any seed likely to be used for the

TABLE II

Optimum Seed Rate for Different Sizes of Seed

Seed	*Mean Size mm.*	*Optimum Rate*	*Surface Area Sq. cm.*	*No. Nuclei*	*Rate of Crystallization*
Through 35, on 48	0.375	20.0	2150	280,000	0.0036
Through 48	0.250	15.0	2400	789,000	0.0033
Morton's icing	0.050	5.5	2800	10,500,000	0.0030

purpose in a sugar factory. Referring to Table II, the efficiency of through 48 seed per unit area is 2150/2400=89.6 per cent. The E.S.A. is then the surface area of a single crystal multiplied by its efficiency. Values for intermediate sizes of grain were calculated and the results are assembled in Table III.

TABLE III

Effective Surface Areas of Different Sizes of Seed

Size mm.	*Surface Area sq. mm.*	*Efficiency*	*E.S.A. sq. mm.*
0.05	0.026	76.8	0.020
0.10	0.041	79.6	0.033
0.15	0.086	82.7	0.071
0.20	0.179	86.0	0.154
0.25	0.273	89.6	0.245
0.30	0.399	93.5	0.373
0.35	0.545	97.7	0.532
0.375	0.626	100.0	0.626

In the factory experiments it was not possible to follow exactly Webre's specification (5) because the pan is not equipped with a mechanical circulator. But the aforementioned magma-mixer (1) appeared quite suitable. The molasses was boiled in the pan until the concentration was such that on striking into the magma-mixer the desired supersaturation was obtained. The stirrers were then set in motion, and the seed added.

Varying seed rates were tested. They were 1, 3, 5 and 50 pounds of Morton's icing sugar per 40 cubic feet molasses or 2.5, 7.5, 12.5 and 125 pounds per 100 cubic feet molasses. The analytical results are presented in Table IV.

The crystal content after 1 hour for the 7.5 lb. seed rate is not reported. The figure obtained by the impurity/water ratio method was not in keeping with the size and number of crystals shown by the photomicrograph. It was therefore concluded that there had been an excessive accumulation of errors in the method in this particular application.

On the basis of size, regularity and number of crystals present, it appears that with the available equipment a seed rate of 12.5 pounds per 100 cubic feet of molasses and an initial supersaturation of 1.50 yield the optimum results. A photomicrograph of the footing after 1 hour's stirring is presented in Figure 1. This, together with the other photomicrographs, was taken on the pan floor immediately after withdrawing the sample.

The factory seed rate equivalent to the optimum of 5.5 per cent. of icing sugar found for the laboratory work (*see* Table II) is 500 pounds per 100 cubic feet of molasses. After the factory experiments at the 12.5 pound seed rate, it was obvious that this would be far too much and the amount was reduced to 125 pounds.

In comparing the results of the different seed rates, it is seen that as the seed rate is increased so the size of the grain after 1 hour decreases and the percentage of conglomerate increases. The increase in the conglomerate count is probably due to the extreme difficulty experienced in avoiding the formation of small lumps in a large mass of finely divided icing sugar. Thus it appears that the majority of the conglomerates were introduced and not formed during stirring. Webre has claimed that the seeding process materially reduces the amount of conglomerate but this is contrary to our experience. The seeding of a 60 purity molasses in a Webre pan has been witnessed at C. Mercedita, Puerto Rico. The amount of conglomerate present was, at the time of the observation, very large. This may have been due to an inefficient mill for grinding the seed but such was not the case during the

TABLE IV

Seeding 60 Purity Molasses. Experimental Factory

Seed rate lbs/100 cu. ft. molasses ...	2.5	7.5	12.5	12.5	125.00
Initial Supersaturation	1.46	1.51	1.50	1.30	1.38
Per cent. Crystal content after 1 hour	20.83	—	18.40	13.08	15.30
Average Crystal size after 1 hour mm.	0.099	0.102	0.098	0.084	0.074
Per cent. Conglomerate	25.0	26.0	33.3	30.00	65.00

experiments conducted in the Experimental Factory. Conglomeration thus appears to be almost inevitable when a large mass of seed is added to a pan.

A comparison of the two runs at the 12.5 pound seed rate and different supersaturations confirms the earlier work (1) that the higher the supersaturation the more rapid the rate of crystal growth.

CIRCULATION

The rate of circulation could be varied in the laboratory but not in the factory. Three stirrer speeds were available, viz. 1.0, 5.0 and 25.0 r.p.m. The optimum seed rate of 5.5 per cent. of Morton's icing sugar was used and the initial supersaturation of the mother liquor was 1.55. The results are graphically presented in Figure 2. Inspection shows that increasing the speed of the stirrers above 5.0 r.p.m. leads to little material increase in the weight of sucrose deposited per 100 grams of massecuite per hour. This observation may be explained by the following: if the stirrers are revolving at such a speed that there is little relative motion between the seed crystals and the mother liquor, then sucrose deposition takes place largely by diffusion. The speed at which the maximum rate of deposition will occur is that which creates the maximum relative motion between the crystals and the mother liquor. Thus the optimum speed at which to operate the stirrer during the process of establishing the fragments will depend upon the design of the stirrer and of the pan, the viscosity of the molasses, the size of the fragments and other factors. The optimum speed for the laboratory conditions is 5.0 r.p.m.

Gomez' Modification of Webre's System

Gomez has described (3) a modification of Webre's system in which the 60 purity molasses is grained spontaneously and not seeded. He retains many of the operating advantages of Webre's system without the necessity for equipping the vacuum pan with a mechanical circulator. Furthermore, the type of crystal formed by graining 60 purity molasses is an extremely attractive one from the sugar boiling point of view and is comparatively free from conglomerate. The resulting crystals are regular in size and the required number appears rapidly because of the high supersaturation necessary. But the process, like all those of graining in the pan, requires a quick decision by the pan boiler as to when to introduce the check charge. There is no absolute control on the number of grain as in the Webre system.

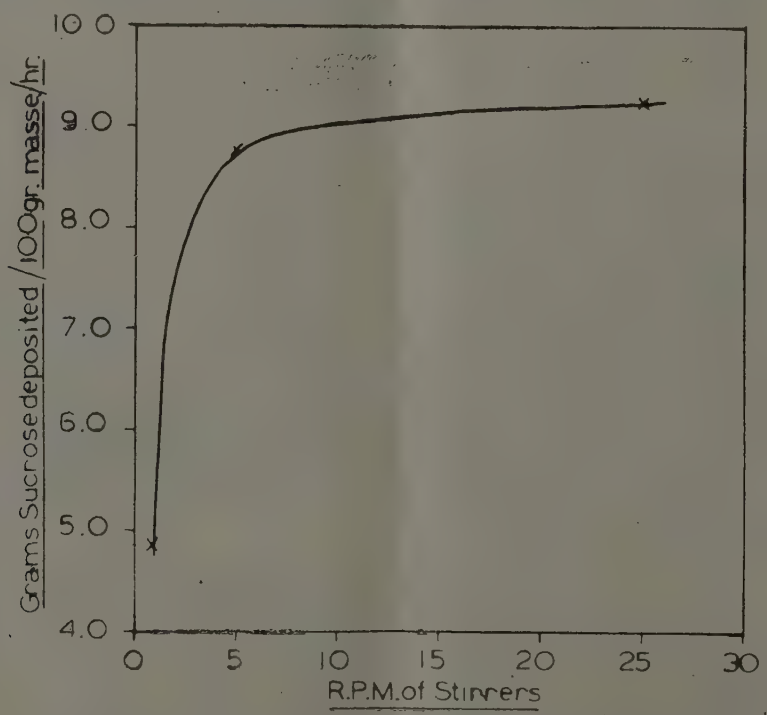

FIG. 2.—Effect of rate of stirring on the sucrose deposition from 60 sucrose/refractometer purity molasses.

The New Graining Technique

Webre's seeding system or Gomez' modification of it have been witnessed or conducted by us in Trinidad, Puerto Rico, Barbados and British Guiana. On analysis, it becomes apparent that apart from the operating features which the two processes have in common, the main advantage of Webre's system lies in the ability to control the number of crystals in the final massecuite. Further, an interesting feature of Webre's system is the maintenance of a constant water content in the mother liquor immediately following the introduction of the seed. It is important, theoretically, that this condition should be observed, because very little surface area is available at that stage for sucrose deposition. If evaporation was to be continued, the upper limit of the metastable zone might be exceeded and fresh nuclei would form. The advantage of adding seed would then be nullified. The use of adequate control instruments can overcome this. Those used at C. Mercedita, Puerto Rico have been described by Mayoral (4).

The main advantage of Gomez' modification is the more desirable type of crystal produced and the comparative freedom from conglomerate. Also, the pan need not be equipped with a mechanical circulator. But the control of the number of nuclei still depends on the skill of the pan boiler.

An ideal system then would be one giving absolute or easier control of the number of nuclei formed; the nuclei should be of a desirable type; control of the process should be facilitated; and expensive equipment should be eliminated as far as possible.

By definition, when molasses is concentrated so that the labile zone of supersaturation is established, crystals of sucrose appear without any further reduction in the water content. This is the basis of the old string proof method, in which low purity molasses are grained at rest in large concrete compartments. But if higher purity molasses are used and grain formation is accelerated by stirring, the type of grain formed is similar to that produced in the vacuum pan by the Gomez' modification. Furthermore, since no evaporation is taking place the water content is constant and the sucrose solubility relationships are not disturbed. The process can therefore be conducted at any predetermined supersaturation value without fear of causing the widely fluctuating conditions such as occur when graining in a vacuum pan.

Basic Specification

The new technique therefore consists of concentrating a molasses of a desirable purity (60-70 purity) in the pan until the labile zone of supersaturation has been established. It is then struck to a lagged vessel equipped with stirrers which are capable of producing vigorous circulation. If the supersaturation value has been properly chosen, grain will form spontaneously, or shock may be added if desired. The formation of crystals will cease as soon as the supersaturation of the mother liquor enters the metastable zone. This occurs in a relatively short time due to the high rate of deposition. While still in the stirring vessel, the crystal nuclei are grown by allowing the mother liquor to cool. The footing, when ready for the pan, contains regular, well-formed crystals of 0.10 to 0.18 mm. in size depending on the purity and other factors. The entire operation is thus conducted at a constant water content, and without utilizing a valuable and expensive unit such as a vacuum pan with mechanical circulator.

Trials in the Experimental Factory

An extensive series of laboratory tests were first conducted on 60, 65 and 70 purity molasses. The primary purpose was to determine whether sufficient crystal nuclei could be formed spontaneously by stirring at constant water content and constant temperature. Varying degrees of supersaturation of the mother liquor were tried. Some of the results for 60 purity have been reported elsewhere (1). The laboratory experiments were entirely satisfactory. It was therefore decided to develop the process in the Experimental Factory.

Accordingly, in the first series, some 60 purity molasses was concentrated in the pan so that a supersaturation of 1.71 would be established after discharge to the stirring vessel. The stirring vessel used was the previously mentioned (1) lagged, open magma-mixer. After discharge the molasses was grain-free when examined at a magnification of 300 ×. The stirrers were set in motion. Grain appeared spontaneously after a few minutes. The experiment was repeated at the same purity and supersaturation, but in this case shock was added in the form of a few ounces of Morton's icing sugar. A third experiment, involving spontaneous granulation, was then conducted at the same purity but at supersaturation 1.84. The results of the three experiments are presented in Table V.

TABLE V

Spontaneous and Shock Granulations at Constant Water Content 60 Purity. Experimental Factory

Type	*Spontaneous*	*Shock*	*Spontaneous*
Initial Supersaturation	1.72	1.71	1.84
Crystal content after 1 hour	35.15	29.83	51.35
Average Crystal size after 1 hr. mm.	0.09	0.09	0.12
%Conglomerate ...	0.00	20.0	16.00

Each of these three granulations were of the desirable molasses type of grain, such as is produced in the pan by the Gomez' modification. The crystal contents after 1 hour are probably exaggerated due to the use, as stated before (1), of the impurity/water ratio method of calculation.

But they are nevertheless comparable. Photomicrographs of the three footings after 1 hour's stirring are presented in Figures 3, 4 and 5. It will be noticed that the crystals formed by the spontaneous method (Figure 3) are more uniform than those produced by the shock method (Figure 4) at the same supersaturation value of 1.71, and that the spontaneously grained footing is free from conglomerate.

The footing produced by spontaneous granulation at the 1.84 supersaturation level (Figure 5) was by far the best seen up to this stage. It was materially better than the seeded footing, see Figure 1. The crystals after 1 hour's stirring were larger and more uniform and the amount of conglomerate was reduced. No checking operation as such had been carried out. The crystals had simply been allowed to form under the conditions of constant water content and efficient circulation. During the hour, the temperature had decreased 4°F below the striking temperature of 154°F.

Need for a More Rapid Growth of Crystal

In operating the Webre system or Gomez' modification, it has been found that in using 60 purity molasses for seeding or graining, a considerable amount of boiling and cutting is necessary before the crystals are of sufficient size for a low grade massecuite footing.

Williams, at Plantation Rose Hall, British Guiana, has overcome this by graining 65 purity molasses, thus retaining the desirable crystal type, and building on syrup so that the footing massecuite is of 72 purity.

Gomez (3) is of the opinion that for practical sugar boiling purposes and with ordinary vacuum pan equipment, the average size of crystal in the finished footing should be 0.15-0.20 mm. This will yield a crystal size at the completion of the "C" strike of 0.30-0.35 mm., and a curing size after cooling in the crystallizer of 0.40-0.45 mm.

Inspection of Table V shows that none of the 60 purity granulations by the new technique after 1 hour's stirring has an average crystal size approaching the required 0.15-0.20 mm. In attempting to achieve Gomez' standards there were therefore two alternatives. They were: (i) To accept the fact that if this new technique is to succeed, then it will have to suffer from the disadvantages of boiling and cutting such as have to be carried out with the Webre system or which Gomez' modification, or (ii) To attempt, without disrupting the desirable straight through flow of molasses in the Webre system, to produce a bigger crystal after 1 hour's stirring. Since the time taken to boil the low grade strikes varies in commercial practice from 3.5-14.0 hours, a one hour stirring period would be well within the required time limits.

The disadvantages of boiling and cutting in the preparation of a footing are that more than one receiver has to be installed in which to store the various stages of cutting or more than one pan has to be used for the low grade strikes. In the larger factories, the latter alternative can be carried out fairly easily but in the majority of medium or small sized factories it would lead to disruption of the pan floor programme. The number of cuts necessary, which is the factor controlling the number of final strikes boiled from a granulation, can in turn be controlled by the number of crystals or amount of seed taken in the original operation. But a great deal of skill is necessary on the part of the pan boiler when a few crystals or a small amount of seed are used, especially during the early stages of the process. The Webre system at C. Mercedita, Puerto Rico, produces 30 cubic feet of final massecuite from each cubic foot of original seeded molasses. Gomez' data (3) show the production of 34 cubic feet of final massecuite per cubic foot of original grained molasses. Both figures indicate considerable preparation of the footing before a low grade strike can be started.

The easiest way to produce a bigger crystal after 1 hour's stirring by the new technique is to increase the purity of the mother liquor. Alternatively, Williams' procedure could be modified and adopted. Of these the first appears to be better adapted to the purpose in view. The next requirement was, therefore, to determine the highest purity at which the typical and desirable molasses grain would appear.

Factory Experiments on the Acceleration of Crystal Growth

Experiments with higher purity mother liquors were therefore conducted in the Experimental Factory using the new technique and the same equipment as that previously described for the 60 purity work.

Molasses of 70 purity were concentrated in the pan and struck to the lagged magma-mixer so that the supersaturation was then 1.67. No shock was added and the grain appeared spontaneously. The results are recorded in Table VI.

TABLE VI

Spontaneous Granulation at Constant Water Content 70 Purity. Experimental Factory

Initial Supersaturation	1.67
Crystal Content after 1 hr.	42.06
Average Crystal size after 1 hr. mm.	0.18
% Conglomerate	0.00

Figure 6 shows the photomicrograph of the footing after 1 hour's stirring. The crystal content is lower than that obtained in the 60 purity series because fewer nuclei were formed. The average size of the crystals after 1 hour's stirring is 0.18 mm. This is within the range specified by Gomez. Also, inspection of the photomicrograph shows that the desirable type of grain had been formed.

From the point of view of the boiling programme and the purity control thereof, there should be little objection to the use of a 70 purity mother liquor for the footing of the low grade strikes. It would not seriously disrupt the straight through flow of the Webre system and it does, as shown, produce a crystal of sufficient size without the necessity of cutting and of storing the cuts in receivers.

FIG. 1.—Footing produced by Webre's method 12.5 pounds seeds/100 cu. ft. added to 60 sucrose/refractometer purity molasses at 1.50 supersaturation. 1 hour's stirring. 50×.

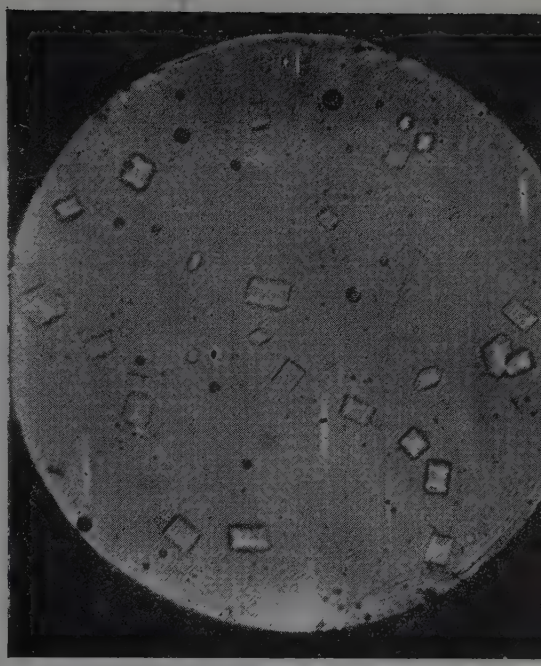

FIG. 3.—Footing produced by the new techniqu 60 sucrose/refractometer purity molasses a 1.72 supersaturation grained spontaneousl 1 hour's stirring. 50×.

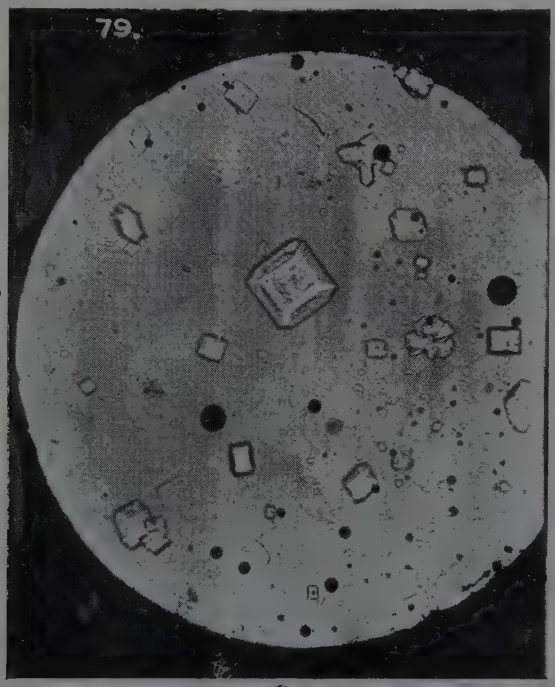

FIG. 4.—Footing produced by the new technique. 60 sucrose/refractometer purity molasses at 1.71 supersaturation grained by shocking. 1 hour's stirring. 50×.

FIG. 5.—Footing produced by the new technique. 60 sucrose/refractometer purity molasses at 1.84 supersaturation grained spontaneously. 1 hour's stirring. 50×.

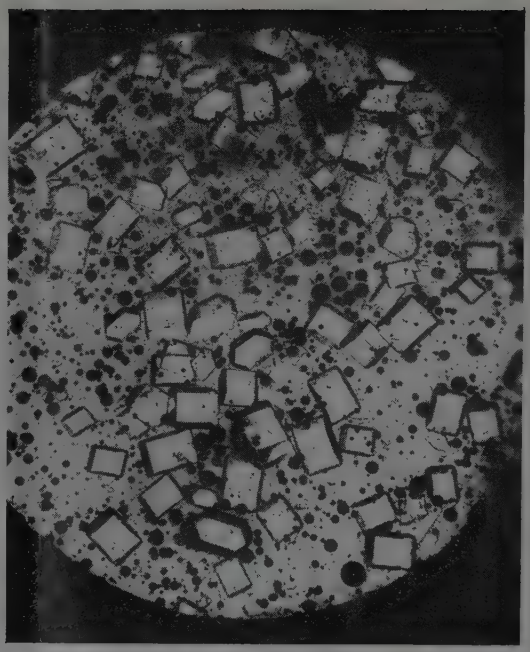

FIG. 6.—Footing produced by the new technique. 70 sucrose/refractometer purity molasses at 1.67 supersaturation grained spontaneously. 1 hour's stirring. 50×.

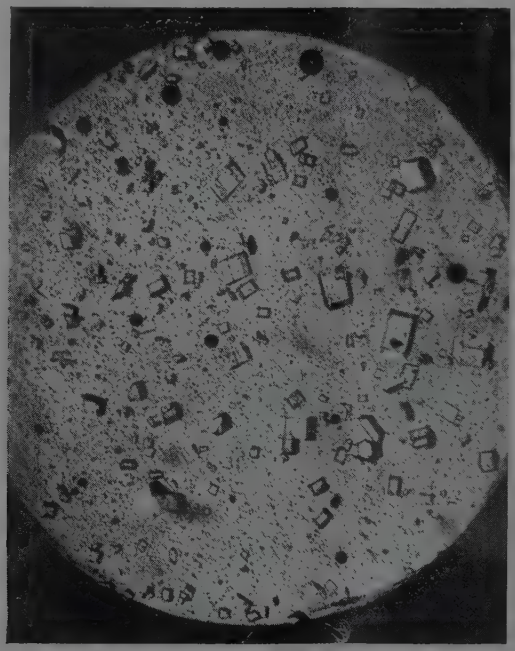

FIG. 7.—Footing produced by graining spontaneously in the pan and checking. 80 sucrose/refractometer purity syrup. 50×.

In order that a comparison may be made between the results obtained with the new technique and with the Java system of graining syrup, Figure 7 reproduces a photomicrograph of a typical graining by waiting of 80 purity syrup in the pan of the Experimental Factory. It is a typical irregular granulation, common to most factories following this practice.

PRACTICAL POSSIBILITIES OF NEW TECHNIQUE

Any worthwhile advance in the technique of pan boiling and crystallization must be based upon a full understanding of the solubility relationships which exist between sucrose and the impurities in solution and upon a knowledge of the effects of these relationships on nuclei formation and sucrose deposition. A fundamental requirement is therefore a rapid method for obtaining the solubility data such as has been described (2). This method is based upon the use of the saturascope and the refractometer. It is simple enough to be placed in the hands of an intelligent native technical assistant. The use of the projection refractometer, such as the model made by Bellingham & Stanley, London, England, further simplifies the procedure and it is also ideal for use on the pan floor. Thus, since the success of the new technique depends largely upon adequate control of supersaturations, the necessary data are easily obtainable.

As a practical guide requiring only a microscope (50× and 300×) and a refractometer, it may be mentioned that most of the molasses so far encountered grain spontaneously in the pan at about 1.80-1.85 supersaturation for 60 purity and 1.70-1.75 supersaturation for 70 purity. The concentration at which this occurs can then be noted from the refractometer. If the new technique is to be subsequently used, allowance must be made for the effect of the temperature drop between the pan and the clean stirring vessel.

When the more desirable procedure of actually determining the solubility data by the cited method is adopted, it will be found in practice that the data need only be checked periodically depending on the uniformity of the cane supply. At the Experimental Factory in Trinidad, it has been observed that the saturation-concentration-temperature line on the solubility graph moves to right or left from one year to the next but that the angle of slope changes little or not at all. Adequate control of the process by the suggested method is a practical proposition.

It is envisaged that by employing the new technique, the graining process can now be placed on a proper, scientific basis. It can be put in the hands of a trained laboratory assistant with the necessary equipment. The only requirement of the pan boiler is to strike the mother liquor at the point determined by the laboratory assistant into the clean stirring vessel. It must be emphasized that the stirring vessel must be properly washed out with hot condensate before each granulation. In practice, there is no reason why the pan boiler should not read the refractometer and the solubility chart to determine the correct point for the transfer. Suitable laboratory assistance must then be provided to determine and periodically check the solubility data.

So far there has not been any need found for checking the granulation in the stirring vessel in a manner similar to that used when graining in the pan. The reason is that since no evaporation is taking place and there is a constant water content, nuclei formation and sucrose deposition takes place at the expense of a decreasing supersaturation in mother liquor. Hence each factory, in accordance with its juice characteristics, type of stirring vessel and other factors, can soon determine the optimum supersaturation at which to grain in order to yield the desired number of nuclei and crystal size.

Depending upon the relationship in volume of the stirring vessel and of the footing required for the low grade pan, more than one footing may be prepared at a time. Alternatively, the footing may be left for a period longer than 1 hour. In either case the crystals will grow due to the deposition of sucrose on cooling. But should this course be adopted, then the supersaturation of the cooled mother liquor must not be allowed to decrease so far that, on taking the footing into the pan and heating it to the operating temperature, the mother liquor becomes undersaturated.

It is possible that a further acceleration of crystal growth and exhaustion of the graining mother liquor might be obtainable if a practical method for checking the granulation in the stirring vessel by heating could be worked out. In such a case, the supersaturation at which the original granulation is to be conducted would have to be increased. Some experiments along these lines are now being carried out.

Acknowledgments

The greater part of the actual experimental work described in this paper formed research problems in successive years for several Fourth Year sugar technology students. But for their interest, loyalty and assistance the work would have been greatly retarded. Acknowledgment is therefore due to Messrs. W. R. Davies (1939-1940), C. L. W. Bradshaw (1941-1942), C. W. Fitzwilliam (1941-1942) and D. L. Harrison (1942-1943) for the very important parts which each played in the development of this new graining technique.

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THE DIAGNOSIS OF SWOLLEN-SHOOT DISEASE OF CACAO

BY

A. F. POSNETTE

(*Botanist, Department of Agriculture, Gold Coast*)

Now that the virus causing swollen-shoot disease of cacao is known to be more widespread on the Gold Coast than was hoped at first, it is important that everyone likely to have the opportunity of inspecting cacao farms should carry with him a mental picture of the symptoms of the disease. This virus, which may be spreading from indigenous trees related to *Theobroma*, causes one of the most serious diseases known to attack cacao, and certainly the most serious in West Africa. Its absence from Nigeria is surprising, and judging from its history and distribution on the Gold Coast, the prospects of Nigerian cacao maintaining its apparent immunity are not reassuring. The disease does not appear to be induced by any particular environment; certainly it has done most damage in a comparatively deforested area of the Eastern Province, but the occurrence of many outbreaks close to forest reserves and in well-shaded farms in areas of higher rainfall, suggests that this may be mere coincidence. A district which produces a large proportion of Gold Coast cacao is where, by the laws of chance alone, a new disease is most likely to occur. The spread of the virus, once transferred to cacao, was undoubtedly aided by the continuous distribution of mature farms in the Koforidua-Akwadum-Suhum district, where the first outbreak probably occurred.

The symptoms of the disease have been described by Steven (3), Dade (1), Posnette (2) and Voelcker and West (4), but these publications have failed to give a complete picture of the symptomatology of the disease for two main reasons: They did not take into account the different strains of the virus, and they overlooked the basis of the pattern in the leaf chlorosis. This cannot be claimed to be a full or final account of the symptoms of Swollen-shoot, because what appear to be new strains are being found at an alarming rate; although each displays a slightly different virulence and mosaic pattern when discovered, it is premature to describe a new strain until it has been transmitted and its characteristic symptoms repeated under different conditions. In this paper the symptoms will be described collectively, and it must be realised that much of the variation in symptoms is due to a corresponding variation in the virus. In this variation, as well as in the symptoms, there is a remarkable parallel with the peach mosaic complex in North America.

Leaf Symptoms

Mosaic.—Vein-clearing appears to be the principle underlying all leaf chlorosis symptoms of the virus strains studied; the chlorophyll fails to develop along the sides of the veins and to a varying distance from them. This clearing usually makes its first appearance as white flecks, before the leaf changes from pink to green. These flecks superficially resemble those cleared round insect punctures, from which they can be distinguished by the absence of a brown speck in the centre. As the pink fades, other areas are defined which will later become almost transparent or pale yellow, but which retain the pink pigment longer than the rest of the leaf, resulting in a pink mottle on a pale green back-ground. This mottle most commonly takes the form of a network, marking the veins. Usually some transparent streaks also are visible, representing veins from which pigment has already cleared. As the chlorophyll develops and the leaf hardens, the final pattern of the yellow mosaic is formed, varying according to the virus strain and to the interval between infection and the development of this particular leaf.

When flushing occurs soon (4—6 weeks) after infection, only a few fine veins (tertiary and smaller) are cleared; spots, or L or T-shaped streaks result. If flushing is long delayed, secondary veins may also be cleared, but this is usually a later symptom appearing on flushes subsequent to that showing the first sign of infection. Generally, the later the stage of infection when the flush develops, the fewer and larger are the cleared areas. The "feather stage", when the mosaic pattern follows the primary veins and resembles a feather, seems to indicate infection about 9—12 months previously.

The statement by Voelcker and West (4) that "completely healthy-looking leaves are never afterwards produced" should not be taken to infer that all subsequent flushes will always show mosaic. Usually they do, but by no means always; nevertheless, with the virulent strains subsequent flushes nearly always look unhealthy in other ways—the leaves are small and pale, and few to a flush.

The only chlorosis likely to be mistaken for swollen-shoot mosaic is that produced by a soil condition apparently due to the presence of excess phosphate in alkaline solution. The mottle formed in leaves by this factor differs from swollen-shoot mosaic in that the cleared areas are *between* the fine veins, which are not cleared, but outlined in green. Swollen-shoot can cause veins to be outlined in green only as a result of the clearing of all the fine veins leaving chlorophyll only along the sides of the main veins. This deficiency chlorosis is usually accompanied by a characteristic leaf distortion. The leaves are narrower than normal, especially at the base, and have an exaggerated apical point (acuminate);

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they are usually twisted to one side, hence the name "sickle-leaf" is used to designate this condition.

In all experimental transmissions, the pattern of the mosaic has remained fixed in the leaf until masked by the general yellowing of an old leaf. As a general rule, the mosaic is most conspicuous in a young leaf and gradually fades as the leaf ages, sometimes until only close inspection with a practised eye can distinguish the pattern. Mosaic has never appeared after a leaf has hardened; the only symptom noticed in leaves formed prior to infection is a premature yellowing indistinguishable from that caused by drought or age.

The virus frequently causes a crinkle; the areas between the main veins bulge upwards (dorsally), imparting a wavy edge to the leaf. Sometimes this symptom is more severe, and the whole leaf is puckered like a cloth in which the stitching, but not the fabric, has shrunk, the stitches representing the veins.

Another form of distortion is caused by the death of large areas between the main veins, which are then drawn closer together, twisting the leaf. The interveinal areas may turn brown and drop out of the leaf, giving a shot-hole effect resembling insect damage. A variation of this necrosis may occur at the edge or tip of the leaf, giving it a ragged appearance.

A common symptom with all virulent strains is a decrease in the size of the leaf. In many infected plants, leaf-size has dwindled progressively with each flush, until the new leaves were less than a quarter the normal size. Perhaps it is this dwarfing carried to its extreme which results in leaves dying and falling when less than an inch long, soon after the opening of the bud.

Leaf shedding.—It has been noticed frequently that the period when the leaf is changing from pink to green is critical; the leaf may wilt, turn almost white, and fall. Thus the first signs of infection may be missed unless inspections are frequent and a tree once marked as infected may show no symptoms a few days later, except leafless green twigs. As already mentioned, this leaf shrivelling and shedding may occur soon after the bud opens, especially with trees which have been infected for several months.

Pod Symptoms

Dwarfing.—The development of dwarfed and rounded pods has long been recognized as a symptom but appears to be confined to the most virulent form of the disease.

Mottling.—Recently a dark green mottling (pink when developed in direct sunlight) on unripe pods has been confirmed as a symptom; as the pod ripens this mottle becomes green or yellow, but disappears when the pod becomes fully ripe. This has been the first sign of infection in transmission experiments with bearing trees. As the pod is growing continuously it is immediately affected by the virus, whereas a new flush must develop before the leaf-symptoms can be seen. Correct diagnosis of pod-mottle requires some practice, as it is easily confused with thrips damage (not staining) and with the skin irregularities caused by uneven growth of the pod.

Stem Symptoms

Swellings need little description as they are unlikely to be mistaken. No small swelling should be taken as diagnostic if it bears signs of insect damage. A swelling often occurs at the end of a shoot, of which the terminal growing point has been checked or killed. On fan branches swellings usually occur at a node, or occasionally just below one, but this generalisation does not seem to apply to chupons.

Short internodes are caused by the slowing down of growth, and by the reduced vigour of the flushes. Although a symptom of swollen-shoot, a similar effect is produced by heavy Psyllid infestation.

Delayed flushing.—An infected tree usually flushes later than nearby healthy trees. This means, of course, that it may be seen still in the resting stage when others have begun to flush; conversely, it may be seen flushing after normal trees have finished. Healthy cacao trees have a regular rhythm of flushes—at Tafo five every year. An infected tree may sometimes skip one, and can be spotted by the absence of any green, unhardened wood at the tips of the branches, and the presence of an undue proportion of old, lichen-covered leaves.

Die-back needs no description. Cage experiments have shown it to be a symptom of virulent strains, even when the environment is good and insects are absent. Die-back is not necessarily associated with swellings.

Root Symptoms

Swellings are the only known symptoms shown by the roots. They are usually found on the tap-root of seedlings and young infected saplings, and on the main laterals of older trees. Recently swellings resembling nodules have been noticed on the fine fibrous rootlets of infected seedlings, but it is not certain that these were caused by the virus.

Flower Symptoms

No abnormalities have yet been found in the flowers of infected trees. From the analogy with peach mosaic, a mottling, or flecking might be expected. Observations during pollination experiments have indicated that once defoliation has become apparent even pollinated flowers are usually shed soon after opening. Badly affected trees are sometimes seen to be carrying a heavy crop, but it seems likely that this was set before or soon after infection. In the absence of foliage, pods are more conspicuous than usual, leading to an exaggerated estimate of yield.

Field Difficulties

All known symptoms of swollen-shoot disease have now been mentioned, and it remains to place them in their true perspective. They are not of equal value in diagnosis because many can be caused by other factors besides the virus. Damage by one of the many species of insects which attack cacao can cause a form of dieback, leaf necrosis, chlorosis, crinkling and shedding, pod mottle, and stem swellings—but not mosaic. Consequently diagnosis often depends on the whole symptom-picture, or the general impression given to the experienced eye. A typical case of swollen-shoot disease need never be mistaken for anything else because either the swellings or the mosaic are distinctive ; but when capsid damage is severe, only dead leaves may be present, and the swellings may be so scarred that they might have been the result of injury. Under these conditions diagnosis is almost impossible, especially if one is dealing with a strain which seldom produces swellings. In most outbreaks of more than about ten infected trees, diagnostic symptoms can usually be found on one or other of the trees, and in the writer's experience, the infection is usually found to extend to the limit of the "dieback area", even though the immediate cause of the dieback appears to be insect damage. In fact, infected trees make such a poor recovery after capsid attack that an outbreak of swollen-shoot is often first noticed as a patch of trees more severely damaged by *Sahlbergella* than the surrounding cacao.

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THE EFEECTS OF SAWDUST ON PLANT GROWTH

It is stated in an article by L. M. Turk published in the *Michigan Quarterly Bulletin** that many questions are continually being asked regarding the value and use of sawdust for soil-improving purposes. Some of the questions most frequently asked are: Will the addition of sawdust to soil have any harmful effect on the soil or crops? Is sawdust of any value in loosening heavy clay soils? Is sawdust a satisfactory material for increasing the organic content of sandy soils? Can sawdust be used satisfactorily as a mulching material? Is sawdust a good material to use as bedding in stables? What is the fertilizing value of sawdust?

In order to answer some of these enquiries certain experiments were carried out to test the effect of mixing sawdust with soils on the growth of crops under greenhouse conditions. The effects of sawdust on nitrification and the accumulation of nitrates under laboratory conditions, and of a sawdust mulch on the accumulation of nitrates under field conditions were also tested.

The experiments indicate that the depressive action of sawdust on plant growth is the result of a deficiency of nitrates in the soil. The decrease in nitrates comes about through the increase in their assimilation by micro-organisms. The carbohydrate material contained in the sawdust supplies the micro-organisms with food and energy which increases their demand for available nitrogen. Evidence is presented to show that nitrate formation is not prohibited by the sawdust, but that the detriment is a result of the assimilation of nitrates after they are formed. The results, taken as a whole, show that sawdust whether used as a mulch or mixed with the soil depresses yields of non-leguminous crops and that the depression is closely associated with a decrease in available nitrates. Furthermore, the results indicate that the depressive effect is only temporary and, when sawdust is mixed with the soil, the detrimental influence will probably disappear within a year or two.

If sawdust is supplemented with some readily available source of nitrogen, it can be added to soils without fear of harmful effects. In all the greenhouse experiments, nitrogen, whether in the form of dried blood, inorganic nitrogen, or in the form of manure, overcame the detrimental influence of sawdust.

Furthermore, the results indicate that sawdust is a satisfactory material for bedding, in that it would have no detrimental effect on the soil unless added in excess. An excess of straw bedding in manure may likewise cause a temporary depression of available nitrates in the soil.

Data reported by the Ohio Agricultural Experiment Station (1), show that 25 pounds of sawdust has the capacity to absorb 100 pounds of liquid; in this respect, it is about equal to chopped straw and has about twice the absorbing capacity of unchopped wheat straw. As an absorbent of liquid manure, therefore, sawdust is very effective but it does not have the capacity to fix or absorb ammonia nitrogen. A ton of wheat straw used as litter in the stable can fix 4.5 pounds of ammonia nitrogen (1).

Sawdust has virtually no fertilizing value. Its composition with respect to nitrogen, phosphoric acid, and potash obviously will vary

* Vol. 26, No. 1, 1943.

(1) Salter, R. M. and Schollenberger, C. J. Farm Manure. *Ohio Agricultural Experiment Bulletin* 605, 1939.

depending upon such factors as the type of wood from which it came, age of the wood and the degree of rotting. Fresh sawdust will contain, on the average, about 4 pounds of nitrogen (N), 2 pounds of phosphoric acid (P_2O_5), and 4 pounds of potash (K_2O), per ton of air-dry material (1); this is less than one-third the total plant food usually contained in wheat straw. Not only is the plant nutrient supply low in sawdust, but its availability is also low. In the true sense of the word, sawdust cannot, therefore, be considered a fertilizing material.

The principal benefits derived by mixing sawdust with soils or using it as a mulch are largely of a physical nature. Sawdust, when mixed with heavy soils, will tend to loosen them thereby increasing ease of tillage and ease of water penetration. Because of the fact that sawdust has a high water-absorbing capacity, it would increase the water-holding capacity of heavy soils, especially those in a poor physical condition Sawdust will decrease losses due to water erosion because of increased water-holding capacity and ease of water penetration. The structure and aeration of heavy soils may be improved by the addition of sawdust.

The principal benefit to be derived from mixing sawdust with sandy soils is to increase their water-holding capacity.

When sawdust is applied as a mulch, its chief function is to protect the moist soil from the direct rays of the sun, and from direct contact with wind and warm dry air. The mulch may reduce cracking of the soil and, in some instances, limit capillary movement of moisture. The mulch may bring about an increased absorption of rainfall by reducing surface runoff. Furthermore, a sawdust mulch may prevent the running together, and the crusting of the surface soil under the impact of raindrops.

It is evident that sawdust may exert many beneficial effects when mixed with the soil or used as a mulch. In soils adequately supplied with nitrogen or if the sawdust is supplemented with nitrogen when used on soils deficient in this element, it may have many beneficial effects. It is to be emphasized, however, that sawdust is an inactive type of organic matter from the chemical point of view and that its effects are largely physical.

All the evidence presented in this paper indicates that sawdust can be satisfactorily applied to soils containing an adequate supply of nitrogen or if supplemented with sufficient nitrogen to off-set the probable depressive effect on the available nitrate supply in the soil.

The results reported here show that nitrogen added in sufficient quantities to give the sawdust the equivalent of about 2 per cent. nitrogen (225 pounds of $NaNO_3$ or 180 pounds of $(NH_4)_2SO_4$ per ton) will overcome the detrimental effects of the sawdust in soils very low in available nitrogen.

REVIEW

A DICTIONARY OF THE FUNGI

By G. C. Ainsworth and G. R. Bisby. Pp VIII + 359. (London: Butler and Tanner Ltd., 1943.) 20s.

This useful little book, in addition to dealing with the synonymy of some 7000 genera of fungi, contains much other useful information on subjects of general mycological interest, such as spraying, genetics, methods, nomenclature, fossil fungi and biographical notes on well-known mycologists. It can also be used as a glossary of mycological terms. The need for a book of this kind has been felt for some time, and its use should help mycologists and pathologists to avoid many of the mistakes in fungal nomenclature which are so common and up to now unavoidable.

For those who are interested in the subject, it should be noted that much of this book is written in Basic English.

R.E.D.B.

PRESERVATION OF HIDES AND SKINS

Throughout the East African territories the annual spoilage of hides and skins through decomposition is considerable. In the drier pastoral areas there is often unavoidable delay due to distance in getting skins to a hide-drying banda, and many fallen hides also are ruined owing to decomposition proceeding in the banda until such time as the hide becomes too dry. In some of the hot, humid areas it is indeed almost impossible at certain seasons to dry even abattoir hides without their sustaining damage.

As a result of experiments carried out by Dr. Reverberi at the Veterinary Research Laboratory Kabete, the following treatment has shown to arrest putrefaction without affecting the subsequent tanning procedures. The hides are placed as soon as possible in a 0.5 per cent. solution of either sodium fluoride or sodium nitrite and are left to soak overnight. They are then allowed to drain and are dried in the usual way. Hides treated in this way may be turned into leather or, in the case of game skins, may be treated by the usual methods so as to leave on the coat. In the latter case hair-slip is prevented. Both sodium nitrite and sodium fluoride solutions of this strength are of low toxicity compared with arsenic and corrosive sublimate, and there should be little risk in their use by natives.—(*East African Agr. Journal*, IX, No. 2.)

GIFT TO COLLEGE DEPARTMENT OF SUGAR TECHNOLOGY

We are greatly indebted to the Foxboro Company of U.S.A. and to the Neal & Massy Engineering Co., Ltd., of Port-of-Spain, for presenting to the Experimental Sugar Factory attached to the College Department of Sugar Technology, a Foxboro automatic juice weighing equipment which was on loan for test purposes during the past two crop seasons.

CHEMICAL NOTES

(Compiled by the College Department of Chemistry and Soil Science)

Sugar Transformation in the Plant

Studies of the transformations of sugars in the sugar cane plant have been continued in Hawaii using blades and roots detached from the plant, and conducting the tests in absolute darkness. It was found that pure glucose in solution is absorbed by the blades or roots and transformed into sucrose. This reaction is affected by many factors, aeration, for example, as when blades are submerged in solutions of glucose or fructose no sucrose is made unless these solutions are aerated. Aeration is also essential for the conversion of glucose to fructose. "Although air is essential for the formation of cane sugar, some of the most important aerobic enzymes play no part in the process. Cyanide, which inhibits the activity of oxidase, peroxidase and catalase, does not inhibit the formation of cane sugar. Evidence indicates that phosphate is vitally concerned in the formation of cane sugar. It seems that simple sugar must combine with phosphate before cane sugar is made. Two dyes which are known to inhibit the formation of fructose diphosphate have been found to inhibit the formation of cane sugar. Other chemicals which are known to inhibit the enzyme which breaks down fructose diphosphate were found to increase the formation of cane sugar. Fructose diphosphate appears to be a stepping stone in the production of cane sugar by the sugar cane plant."—(*Intern. Sugar Journal*, 1943, Vol. XLV, No. 529, p. 29.)

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Liebig and the Superphosphate Industry

The names of Escher, Liebig, Murray, and Lawes illuminate the pages of superphosphate history. Escher first conceived of treating bones with acid, Liebig made the first superphosphate, Murray patented the process and gave the product its name, and Lawes founded the superphosphate industry. Liebig's name shines brightest because of the brilliance of his conception of the need of crops for inorganic elements as distinct from the long-established concept that humus was the food of plants. He first conceived of the idea of mineral manures and laid the groundwork for the complete fertilizer industry. Originally Liebig was not very enthusiastic about superphosphate, because he thought that water-soluble phosphates would be carried away by drainage water. So he melted his mineral mixtures in huge iron pots to render them insoluble, putting them through a pulverizing mill before they were applied to the soil. Not until the publication of Way's classic researches on the fixing powers of soils did Liebig realize his error, but by that time Lawes was well on the way to success as a producer of superphosphate. The commercial production of ammonium phosphates, ammoniated superphosphates, calcined phosphates, metaphosphates, and organic phosphates has raised a series of new problems, the answers to which must be found before fertilization with phosphates can proceed intelligently. It is interesting to note that, with further investigation of this problem, Liebig's question on the use of water-soluble phosphates, of a century ago, has returned again to plague us.—(*Journ. Soc. Chem. Indus.*, Vol. 59, No. 39, p. 680.)

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Manurial Experiments on Tomatoes

Reports of investigations on the tomato occupy most of the twenty-eighth annual report of the Cheshunt Experimental and Research Station. Trials of the value of muriate of potash as against sulphate of potash resulted in a small, though scarcely significant, increase of crop in favour of the sulphate. No harmful foliage effects were observed when muritate was added, and it would seem that widespread doubts about the suitability of muriate of potash for the indoor tomato crop are now dispelled.

Tomatoes in sand culture exhibited clear-cut symptoms when deprived of boron, manganese and magnesium. Omission of boron and magnesium from the culture solution produced marked stunting, and boron deficiency caused a leaf scorch similar to that sometimes associated with acute potassium starvation. Lack of magnesium induced a withering of the tips of leaflets, whereas the absence of manganese brought about a mottling of the leaf with uniform yellow specks.—(From *Nature*, Vol. 152, No. 3869.)

* * *

Bangalore Method of Composting

Investigations have been carried out which aimed at modifying the hot-fermentation process by the incorporation of new features in such a way as to bring down nitrogen losses to a low level and to promote the progressive humification of the manure. The new process developed as a result of such investigations is styled the Bangalore process and differs from the usual aerobic methods of composting as follows:—

(*a*) the use of an earth-plaster for preventing losses of nitrogen, and

(*b*) the application of the principle of "controlled aerobiosis" of the refuse so as to promote humification of the refuse to the extent necessary for improving the quality of the manure, but not to involve unnecessary loss of humifiable organic matter or of nitrogen.

Extensive tests were carried out with the improved process, and the largest quantity of manure that may be possibly recovered from the original refuse has been obtained in addition to conserving most of the nitrogen. Further features of the process are the low water and labour requirements and consequently low composting charges. —(*Soils and Fertilizers*, VI, No. 3.)